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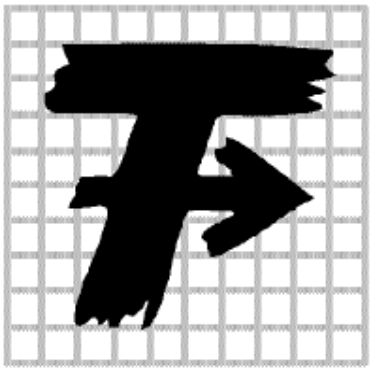

Disertačni práce
Ph.D. Dissertation

Plasma Treatment of Textile Fabrics

2010

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IN THE NAME OF GOD

THE BENEFICENT AND THE MERCIFUL

ABSTRACT

In this project, the application of plasma in textile industry was studied.

For this purpose, four kinds of experimental equipment were used and the application of them in different fields of textile finishing was investigated.

The first one was direct current (DC) Magnetron sputtering. The parameter of this setup (current, voltage, time of exposure, gas type, power, electrode type, …) were changed for different applications. By using this system with different conditions, the hydrophilicity, dyeability, water repellency and antibacterial activity of fabrics have been improved.

The second experimental method was Diffuse Coplanar Surface Barrier Discharge (DCSBD). Also Ion beam were used for improving the chemical and physical properties of Polypropylene (PP).

The Electron gun was used for increasing the dyeability and printability of PP fabrics. This setup was used as a new method for decoration of polymeric fabrics. Also the penetration of electron irradiation on polypropylene films was investigated.

For confirming the achieved results, several instruments were used for analyzing the samples such as: Scanning Electron Microscope (SEM), Fourier Transform Infrared spectroscope (FTIR), Energy Dispersive X-ray (EDX), reflective and absorption spectrophotometer, Atomic force microscope (AFM), light microscope and many textile testing.

The present examples show that plasma technology leads to variety to processes to modify fiber or textile materials to fulfill additional highly desirable requirements. It is to be expected that, this technology, which has been known for a long time and is being used in different branches of industry, in the near future will conquer textile as well.

ABSTRAKT (IN CZECH)

V této práci bylo zkoumáno využití plazmatu pro textilní průmysl. Za tímto účelem byla využita čtyři různá zařízení, jejichž využití v zušlechťování textilií bylo v této práci sledováno.

Jako první zařízení bylo použito DC Magnetron. Nastavené parametry (proud, napětí, doba expozice, typ plynu, výkon, typ elektrody, …) byly měněny pro různé aplikace. Použitím různých podmínek úpravy byla zvýšena hydrofilita, barvitelnost a vodoodpodivost a antibakteriálnbí aktivita textilie.

V druhé aplikaci bylo využito difúzního koplanárního povrchového bariérového výboje (DCSBD). Dále bylo použito využito svazku ionů pro zlepšení chemických a fyzikálních vlastností polypropylenu.

Elektronové dělo bylo využito pro zvýšení barvitelnosti a kvality tisku polypropylenových tkanin. Toto zařízení bylo využito jako nová metoda pro dekoraci polymerních tkanin. Dále byl sledován průnik elektronového záření polypropylenovými fóliemi.

Pro potvrzení dosažených výsledků byly využity tyto metody: skenovací elektronová mikroskopie (SEM), infračervená spektroskopie s Fourierovou transformací (FTIR), rentgenová analýza, remisní a absorpční spektrofotometrie, mikroskopie atomárních sil (AFM) a světelný mikroskop a řada textilních zkoušek.

Prezentované výsledky dokazují, že technologie plazmatu vede k různým postupům modifikace vláken či tkanin s cílem dosáhnout požadovaných vlastností. Je třeba počítat s tím, že tato technologie, která je známa již dlouhou dobu a je využívána různými odvětvími průmyslu, v blízké budoucnosti dobude také textilní průmysl.

Dedicated to
my Mother,
Fakhri Yabandeh, who is a constant
source of encouragement

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Liberec, Czech Republic, 2010

Sheila Shahidi

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Preface

This dissertation is submitted to the Faculty of Textile in partial fulfillment of the requirements for the degree of Doctor of Philosophy at the Technical University of Liberec, Czech Republic. It is prepared in six chapters. Chapter one contains introduction to surface modification methods. In Chapter 2, the fundamental of plasma is discussed and in Chapter 3, some basis about plasma technology in textile industry is explained.

Methods and materials used in this study are described in chapter four. As is mentioned four types of plasma systems was used in this project and several instruments were used for analyzing the results. Chapter five demonstrates the obtained results. In this project, application of plasma in different fields of textile finishing was investigated and these applications are discussed separately.

Overall conclusions of this dissertation are presented in chapter six. And at the end the list of related patents and publication are presented.

1- INTRODUCTION

1-1. Introduction to Surface Modification

Modification is used to designate a deliberate change in composition or structure leading to an improvement in some fiber properties.

The aims of modification can be:

- To obtain new properties
- To enhance some positive properties
- To suppress some negative properties, such as reducing the pilling tendency, increasing the absorbing capacity, etc.

The basic problem is, however, that there does not exist an ideal modification that eliminates all the negative properties and preserves all the positive properties of the fibers. This is why there are a great number of different single-purpose modifications. [1]

In spite of the great number of existing modification methods no consistent classification is available as yet. Some authors divide the methods into two groups depending on whether they involve changes in fiber composition (chemical modification) or changes in fiber structure (physical modification).

Surface modification of polymers has become an important research area in the plastic industry. Many polymers have very good bulk properties and are in-expensive, but many industrial applications such as adhesion, biomaterials, protective coatings, friction and wear, microelectronic devises, thin-film technology, and composites require these polymers to have special surface properties. Because polymers are inert materials and usually have a low surface energy, they often do not possess the surface properties needed to meet the demands of various applications. Advances in surface treatment have been made to rather chemical and physical properties of polymer surfaces without affecting bulk properties. Technologies such as surface modifications, which convert inexpensive materials into valuable finished goods, will become even more important in the future as material cost becomes a significant factor in determining the success of an industry. [2] Some of these methods are discussed as bellow:

1-2-Flame Treatment:

Flame has been used to treat polyolefin and other polymeric products. The setup-a burner and fuel tank is very simple and portable. However, craftsmanship is needed to produce consistent results. Typically, flame treatment produces an oxidized polymer surface for improved bond ability with an adhesive or for improved printability and mark permanence. [2]

1-3-Corona Discharge:

Corona discharge, which can be obtained with a relatively simple and inexpensive setup, is a popular industrial technique for the surface treatment of polymeric films. In a corona discharge system, plasma is produce when air is ionized by a high electric field. The atmosphere pressure plasma, which is called a corona discharge, causes various chemical and physical changes on a polymer surface for improved bond ability and printability.

Corona treatment system consists of a high-voltage and high-frequency generator, an electrode, and a grounded metal roll. The grounded roll is usually covered with an insulating material such as polyester, ceramic, epoxy, or silicon rubber. The system can be treated as a large capacitor, with the electrode and the grounded roll as the plates of the capacitor and the roll covering and the dielectric. Corona occurs when a high voltage is applied across the electrodes to cause ionization of air. Plasma is formed, and a light blue color can be observed in the air gap. This atmospheric pressure plasma is called a corona discharge.

Corona discharge has been proved to be a useful method for improving the polymer surface hydrophilic property, especially in the composite material strength improvement, the treatment can improve the surface affinity and the sticking strength with some hydrophilic polymers, because the treatment can lead to the increasing of the high reactive free radical oxygen in the polymer surface. Corona discharge was also applied in the chemical graft copolymerization, this is also based on that after the irradiation, new active group is produced and the monomer can be successfully grafted onto those active groups in the irradiated material. Some other researchers have introduced the corona discharge treatment into the textile surface treatment to improving the wool fabric printability or the wool fabric shrink resistance properties.

Corona discharge technology is also widely used in the printing of some polymer films. The treatment has been confirmed to be simpler and more practical than any other chemical and physical methods because the samples can be quickly treated under atmosphere pressure.

1-4-Chemical Treatment:

Chemical treatment has been used in industry to treat large objects that would be difficult to treat by other commonly used industrial technique such as flame and corona-discharge treatments. Chemical etchants are used to convert smooth hydrophobic polymer surfaces to rough hydrophilic surfaces by dissolution of amorphous regions and surface oxidation. Chromic acid is the mostly widely used etchant for polyolefins and other polymers. [2]

1-5-Ion beam modification:

Ion beams have been used to texturize polymer surfaces, especially fluoropolymers, to increase adhesion. The texture produced on a polymer surface depends on the ion beam energy and beam dose. For example, different types of surface texture are produced at different beam doses. At doses (1.15×10^{15} atoms/cm^2) the PTFE surface for example is covered with a dense network of isolated fissure ranging in size from 3 to 500 μ m. at a higher beam dose (7.5×10^{15} atoms/cm^2) long cracks appear on the surface. At an even higher beam dose (2.3×10^{16} atoms/cm^2), the material splits into individual filaments. Finally at a beam dose of (6.9×10^{16} atoms/cm^2), a dramatic change occurs at the surface. The surface is totally covers with cones. At high beam doses, most ion-etched fluoro polymer surfaces have cone or spike-like features, with greatly increased the surface area available for bonding with the adhesive.

Ion beam modification of polymers has perhaps been most useful in preparing polymer surfaces for metallization, adhesion of titanium films to polyethylene surfaces improved dramatically after polyethylene surfaces had been bombarded with argon ions. The rough texture of the modified surface may provide mechanical inter locking with the metal layer. Surface conductivity of a polymer can also be increased drastically by ion – beam irradiation. At high doses of ion-beam irradiation, a surface layer of graphite-like structure can be formed at various polymer surfaces. [2]

1-6-Surface Grafting:

Radiation grafting is a very versatile technique by which surface properties of a polymer can be tailored through the choice of different monomers. The most common radiation sources are high-energy electrons, γ radiation, and ultraviolet and visible light. Grafting is usually performed by irradiating the polymer in the presence of solvent containing a monomer. Alternatively, grafting can be initiated thermally by contacting the polymer, which has been preirradiated in air to produce reactive groups on the surface, with monomer. Polymer has frequently been modified by graft polymerization to change their physical and chemical properties for such applications as biomaterials, membranes, and adhesives. Grafting can occur at the surface and in the bulk. For surface modification applications, thick grafting layers are unnecessary and even undesirable because they may change bulk physical properties of the polymer such as crystallinity and tensile modulus.

The energy sources most commonly used in radiation grafting are high energy electrons, γ radiation, X-ray, ultraviolet (UV), and visible light. Electron –beam radiation, γ radiation and X-ray are usually classified as ionization radiation. The study of effects produced by UV or visible light is known and classified as photografting. [2]

1-6-1-Surface photografting:

Photografting has been extensively used to improve such properties of polymer surfaces as adhesion, printability, antistatic, antifogging, antistaining, wear ability, biocompatibility, and dyeability. For example, in biomedical applications, various polymer surfaces have been grafted with different monomers to improve biointraction for applications entailing sell adhesion, biocompatibility, and immobilization of enzymes and bio active materials. Photografting has been used to modify electrostatic properties of polymers. Surface photografting can be performed in vapor or liquid phase.

A polymer film is fed continuously in to a chamber containing a solution of monomer and initiator. This is referred to as the presoaking step. The film is then fed in to a temperature–controlled reactor, which is filled with nitrogen to keep a positive pressure during the grafting process. The amount of time the film takes to travel from the presoaking solution to the area where it is irradiated with a UV lamp is controlled,

allowing the diffusion of the monomer and initiator in to the amorphous regions of the polymer. The irradiation time is regulated by varying the motor speed. The most important feature in this process, which differentiates it from other processes, is the presoaking step. With the presoaking step, grafting times can be as short as 5-10 seconds.

The structure of the polymer substrate has been found to affect the grafting rate. Grafting occurs only in the amorphous regions of the polymer because high crystallinity prevents penetration of the reaction solution in to the base polymer. The grafting rate for high-density polyethylene is slower than that of the low-density polyethylene because of the higher crystallinity of the former polymer. The grafting rate and the properties of a grafted surface are strongly related to the properties of the solvent.

Also surface photografting can be performed by exposing a preirradiated surface to a monomer. It is known that peroxides, which are formed at various polymer surfaces by UV irradiation, can be used to initiate grafting copolymerization. First the polymer is irradiated by a UV lamp in air. Then the irradiated film is put into a reactor containing a deaerated monomer solution, which is kept at an elevated temperature. Decomposition of the peroxides at high temperatures initiates surface grafting. [2]

1-6-2- Surfaces grafting with ionization radiation:

Ionization radiation such as high-energy electrons, X-rays, and γ-rays can be displace electrons from atoms and molecules, producing ions. It differs from other types of radiation such as infrared, visible, and UV in that it is highly energetic and delivers to the irradiated material a large amount of energy, much greater than that associated with chemical bonds. Common industrial ionization radiation sources are high-energy electrons (0.1-10 MeV) and cobalt-60 sources, and γ radiation.

Electron beams from 0.1 to several mega electron volts are used for high doses and high speeds in various industrial processes, with penetration up to several millimeters for polymeric materials. [2]

1-6-3-Surface grafting with pretreated polymer surfaces:

In addition to UV, high energy electron, and γ ray irradiation, various methods, including plasma treatment, corona treatments, and ozone exposure, are commonly used to produce peroxides on polymer surfaces for surface grafting. Peroxides generated by UV, high-energy electron and γ ray irradiation of a polymer film, in general, can be found deep beneath the surface, whereas those created by plasma treatment, corona discharge and ozone exposure are usually confined to the surface. [2]

1-7-Photon Irradiation:

Surface modification by UV and IR lasers is useful in some specific applications. One key advantage of laser treatment is that the area to be treated can be very small and localized. Depending on the level of power chosen, ablation or chemical and physical changes can occur.

Various chemical changes occur on photon-irradiated polymer surfaces. When PTFE was irradiated with ArF laser at high fluencies, defluorination and surface oxidation occurred.

For polypropylene, formation of oxygen functional groups such as C-O and C=O groups was detected after UV laser irradiation in air and water, and in ozone. The treated surfaces were shown to have improved bond ability with an epoxy adhesive. The surface of poly(vinyl chloride) becomes electrically conductive after successive UV irradiation in chlorine and nitrogen and argon laser irradiation in air. [2]

1-8-Plasma Treatment:

A typical plasma system consists of a gas inlet, a reactor vessel, a vacuum pump, a matching network, and a power source. Various reactors have been used in plasma processing for dc and low-frequency glow discharges, internal electrodes are necessary.

A typical reactor is a bell jar with circular or square electrodes. As the frequency increases, electrodes maybe placed out side the reactor vessel. Plasma treatment has been used to improve print ability, wettablility, bond ability, biocompatibility, surface hardness, and surface heat resistance. It is also a means of cleaning polymer surfaces without a solvent and of introducing cross-linking at the surface.

The different types of gas or mixtures of gases that can be used for plasma treatment of polymers include argon, helium, hydrogen, nitrogen, ammonia, nitrous oxide, , oxygen, carbon dioxide, sulfur dioxide, water, and tetrafluoromethane. Oxygen and oxygen-containing plasmas are the most frequently and are very effective at increasing the surface energy of polymers. Nitrogen and nitrogen-containing plasmas are used to produce nitrogen functionality such as amino groups on polymer surfaces. Fluorine and fluorine-containing plasma are used to decrease the surface energy and to increase the hydrophobicity of polymer surfaces. Each gas produces a unique modified surface. In an oxygen plasma two process occur simultaneously: etching of polymer surface through the reaction of atomic oxygen with the surface carbon atoms, giving volatile reaction products; and the formation of oxygen functional groups at the polymer surface through the reactions between the active species from the plasma and the surface atoms. The balance of these two processes depends on the operation parameters of a given experiment.

Many parameters such as the nature of polymer substrate, the temperature of the substrate, electrode materials, pressure, power level, and gas flow rate play a significant role in affecting the outcome of a plasma treatment. [5]

2-THEORY

2-Plasma

Plasma is by far the most common form of matter. Plasma in the stars and in the tenuous space between them makes up over 99% of the visible universe and perhaps most of that which is not visible.

Figure 2-1: plasma in thunder

On earth we live upon an island of "ordinary" matter. The different states of matter generally found on earth are solid, liquid, and gas. We have learned to work, play, and rest using these familiar states of matter. Sir William Crookes, an English physicist, identified a fourth state of matter, now called plasma, in 1879. Plasma temperatures and densities range from relatively cool and tenuous (like aurora) to very hot and dense (like the central core of a star). Ordinary solids, liquids, and gases are both electrically neutral and too cool or dense to be in a plasma state. The word "PLASMA" was first applied to ionized gas by Dr. Irving Langmuir, an American chemist and physicist, in 1929.vapor discharges, and he noticed similarities in their structure - near the boundaries as well as in the main body of the discharge. While the region immediately adjacent to a wall or electrode was already called a "sheath," there was no name for the quasi-neutral stuff filling most of the discharge space. He decided to call it "plasma."
While his relating the term to blood plasma has been acknowledged by colleagues who worked with him at the General Electric Research Laboratory, the basis for that connection is unclear. One version of the story has it that the similarity was in carrying particles, while another account speculated that it was in the Greek origin of the term,

meaning "to mold," since the glowing discharge usually molded itself to the shape of its container. In any case, it appears that the first published use of the term was in Langmuir's "Oscillations in Ionized Gases," published in 1928 in the proceedings of the National Academy of Sciences.

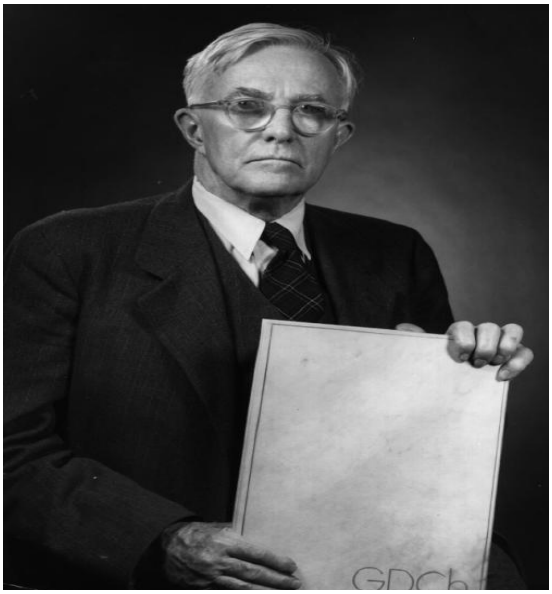

Figure 2-2: The photo of Dr. Irving Langmuir

Thus the term "plasma" was first used to describe partially (if not weakly) ionized gases. The term plasma apparently did not find immediate widespread use in the scientific community. It did eventually catch on, however, but in some cases the term was inappropriately limited to highly ionized gas [3,4].

2-1-What is Plasma?

In physics and chemistry, a plasma is typically an ionized gas. Plasma is considered to be a distinct state of matter, apart from gases, because of its unique properties. "Ionized" refers to presence of one or more free electrons, which are not bound to an atom or molecule. The free electric charges make the plasma electrically conductive so that it responds strongly to electromagnetic fields. Plasma typically takes the form of neutral gas-like clouds (e.g. stars) or charged ion beams, but may also include dust and grains (called dusty plasmas). They are typically formed by heating and ionizing a gas, stripping electrons away from atoms, thereby enabling the positive and negative charges to move more freely. In an ordinary gas each atom contains an equal number of positive

and negative charges; the positive charges in the nucleus are surrounded by an equal number of negatively charged electrons, and each atom is electrically "neutral." A gas becomes a plasma when the addition of heat or other energy causes a significant number of atoms to release some or all of their electrons. The remaining parts of those atoms are left with a positive charge, and the detached negative electrons are free to move about. Those atoms and the resulting electrically charged gas are said to be "ionized." When enough atoms are ionized to significantly affect the electrical characteristics of the gas, it is a plasma [1,2 and 3]. Plasmas are radically multi scale in two senses (1) most plasma systems involve electro-dynamic coupling across micro-, meso- and macro scale and (2) plasma systems occur over most of the physically possible ranges in space, energy and density scales. The figure here illustrates where many plasma systems occur in terms of typical densities and temperatures.

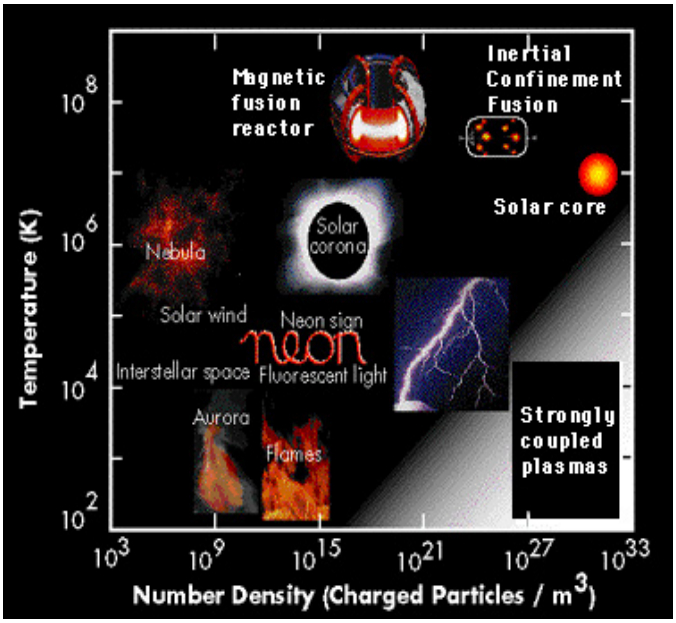

Figure 2-3 :The figure here illustrates where many plasma systems occur in terms of typical density and temperature conditions. Plasma temperatures and densities range from relatively cool and tenuous (like aurora) to very hot and dense (like the central core of a star). Ordinary solids, liquids, and gases are both electrically neutral and too cool or dense to be in a plasma state.

Plasma consists of a collection of free-moving electrons and ions - atoms that have lost electrons. Energy is needed to strip electrons from atoms to make plasma. The energy can be of various origins: thermal, electrical, or light (ultraviolet light or intense visible

light from a laser). With insufficient sustaining power, plasmas recombine into neutral gas. Plasma can be accelerated and steered by electric and magnetic fields which allows it to be controlled and applied. Plasma research is yielding a greater understanding of the universe. It also provides many practical uses: new manufacturing techniques, consumer products, and the prospect of abundant energy.

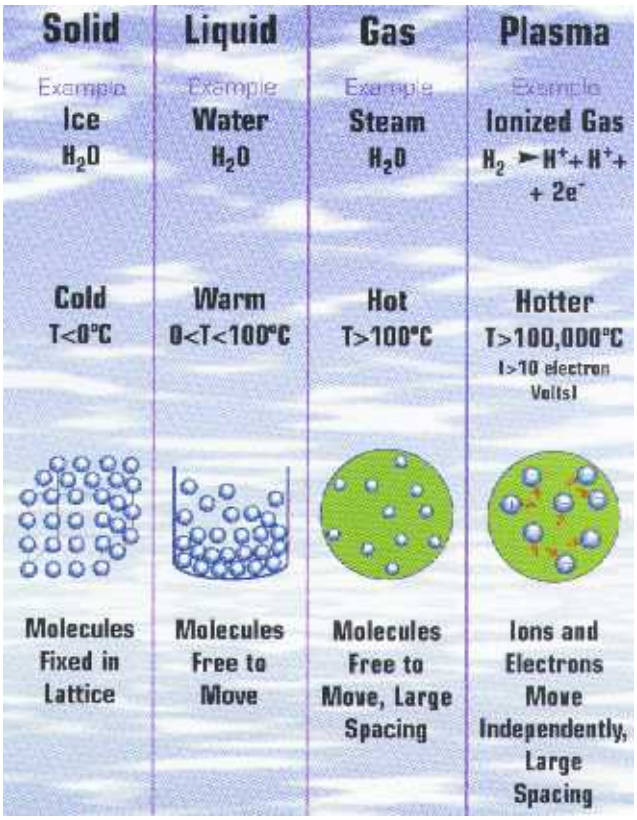

Figure 2- 4: different phase of matter

2-2-Plasma Parameters:

2-2-1-Degree of ionization

For plasma to exist, ionization is necessary. The word "plasma density" by itself usually refers to the electron density, that is, the number of free electrons per unit volume. The degree of ionization of a plasma is the proportion of atoms which have lost (or gained) electrons, and is controlled mostly by the temperature. Even a partially ionized gas in which as little as 1% of the particles are ionized can have the characteristics of a plasma (i.e. respond to magnetic fields and be highly electrically conductive). The degree of

ionization, α is defined as $\alpha = n_i / (n_i + n_a)$ where n_i is the number density of ions and n_a is the number density of neutral atoms. The electron density is related to this by the average charge state $\langle Z \rangle$ of the ions through $n_e = \langle Z \rangle n_i$ where n_e is the number density of electrons.

2-2-2-Temperatures

Plasma temperature is commonly measured in Kelvin's or electron volts, and is (roughly speaking) a measure of the thermal kinetic energy per particle. In most cases the electrons are close enough to thermal equilibrium that their temperature is relatively well-defined, even when there is a significant deviation from a Maxwellian energy distribution function, for example due to UV radiation, energetic particles, or strong electric fields. Because of the large difference in mass, the electrons come to thermodynamic equilibrium among themselves much faster than they come into equilibrium with the ions or neutral atoms. For this reason the **ion temperature** may be very different from (usually lower than) the **electron temperature**. This is especially common in weakly ionized technological plasmas, where the ions are often near the ambient temperature. Based on the relative temperatures of the electrons, ions and neutrals, plasmas are classified as **thermal** or **non-thermal**. Thermal plasmas have electrons and the heavy particles at the same temperature i.e. they are in thermal equilibrium with each other. Non thermal plasmas on the other hand have the ions and neutrals at a much lower temperature (normally room temperature) whereas electrons are much "hotter". Temperature controls the degree of plasma ionization. In particular, plasma ionization is determined by the **electron temperature** relative to the ionization energy (and more weakly by the density) in accordance with the Saha equation. A plasma is sometimes referred to as being **hot** if it is nearly fully ionized, or **cold** if only a small fraction (for example 1%) of the gas molecules are ionized (but other definitions of the terms **hot plasma** and **cold plasma** are common). Even in a "cold" plasma the electron temperature is still typically several thousand degrees Celsius. Plasmas utilized in **plasma technology** ("technological plasmas") are usually cold in this sense.

2-2-3-Electro magnetic Fields:

The Maxwell equations for electromagnetism and the plasma Boltzmann equation are the basic equations for studies of electromagnetic systems of which plasmas are a prime example.

In analysis, plasmas are far harder to model than solids, liquids, and gases because they act in a self-consistent manner. The separation of electrons and ions produce electric fields and the motion of electrons and ions produce both electric and magnetic fields. The electric fields then tend to accelerate plasmas to very high energies while the magnetic fields tend to guide the electrons. Both of these mechanisms, the accelerated (or fast) electrons and the magnetic fields produce what is called ***sychrotron radiation***, so called because it was first discovered in large magnetized containers of electrons beams in laboratories on earth. Because of their self-consistent motions, plasma are rampant with instabilities, chaosity, and nonlinearities. These also produce electric and magnetic fields but also electromagnetic radiation. For example, all beams of electrons produce microwaves. Plasma science has, in turn, spawned new avenues of basic science. Most notably, plasma physicists were among the first to open up and develop the new and profound science of chaos and nonlinear dynamics. Plasma physicists have also contributed greatly to studies of turbulence, important for safe air travel and other applications. Basic plasma science continues to be a vibrant research area. Recent new discoveries have occurred in understanding extremely cold plasmas which condense to crystalline states, the study of high-intensity laser interactions, new highly-efficient lighting systems, and plasma-surface interactions important for computer manufacturing. The term fundamental is used to denote plasma because the constituent components of plasmas, electrons and ions, are the longest lived particles know. Their lifetimes far exceed that of any other known particle. Thus long after other forms of matter and radiation have ceased to exist, it will have reverted back into the plasma state [1-6].

2-2-4-Common Artificial Plasma

Most artificial plasmas are generated by the application of electric and/or magnetic fields. Plasma generated in laboratory setting and for industrial used can be generally categorized by:

1) The type of power source used to generate the plasma; DC, RF and microwave.
2) The pressure at which they operate; vacuum pressure (< 10mTorr), moderate pressure (~ 1 Torr), and atmospheric pressure (760 Torr).
3) The degree of ionization within the plasma; fully ionized, partially ionized.
4) The temperature relationships within the plasma; Thermal plasma (Te = Tion = Tgas), Non-Thermal or 'cold' plasma (Te >> Tion = Tgas)
5) The electrode configuration used to generate the plasma.
6) The magnetization of the particles within the plasma; Magnetized (both ion and electrons are trapped in orbits by the magnetic field), partially magnetized (The electrons but not the ions are trapped by the magnetic field), non-magnetized (the magnetic field is too weak to trap the particles in orbits but may generate Lorentz forces).

2-2-5-Examples of Industrial/Commercial Plasma:

Low Pressure Discharges:

Glow Discharges: Non-thermal plasmas generated by the application of DC or low frequency RF (<100 kHz) electric field to the gap between two metal electrodes.
Probably the most common plasma it is type of plasma generate within fluorescent light tubes.

Capactively Couple Plasmas (CCP): Similar to glow discharges but generated with high frequency RF electric fields, typically 13.56 MHz. It differs from glow discharges in that the sheaths are much less intense. These are widely used in the micro fabrication and integrated circuit manufacturing industries for plasma etching and plasma enhanced chemical vapor deposition.

Inductively Coupled Plasmas (ICP): Similar to a CCP and with similar applications but the electrode consists of a coil wrapped around the discharge volume which inductively excites the plasma.

Atmospheric Pressure

Arc: This is a high power thermal discharge of very high temperature ~10000K. It can be generated using various power supplies. It is commonly used in metallurgical processes. For example it is used to melt rocks containing $Al_2\ 0_3$ to produce aluminum.

Corona: This is a non-thermal discharge generated by the application of high voltage to sharp electrode tips. It is commonly used in ozone generator and particle precipitators.

Dielectric Barrier Discharge (DBD): Invented by Siemans this is a non-thermal discharge generated by the application of high voltages across small gaps wherein a non-conducting coating prevents the transition of the plasma discharge into an arc. It is often mislabeled 'Corona' discharge in industry and has similar application to corona discharges. It is also widely used in the web treatment of fabrics. The application of the discharge to synthetic fabrics and plastics functionalizes the surface and allows for paints, glues and similar materials to adhere.

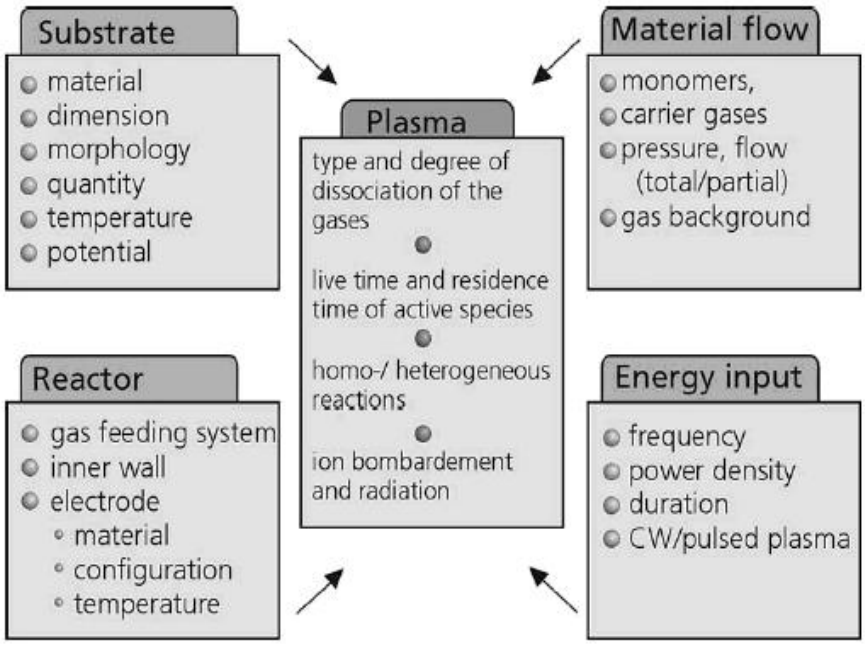

Figure 2-5: Plasma Parameters [7].

2-3-Plasma Chemistry:

Most processing plasmas are created in a molecular gas or a gas mixture that contains molecules. There are therefore a very large number of constituent species. The energy required to remove a single electron from a molecule or atom is known as the ionization

potential. The energies required to break molecules into various constituent parts are called the dissociation energies.

Just as in a neutral gas, the moving charged and neutral particles are colliding or interacting and reacting. The presence of energetic charged particles, particularly the electrons, lead to unique plasma chemistry. There are a huge number of interactions that can take place, particularly in a molecular plasma.

Table 2-1 lists some of the important types of reactions that occur in plasmas where most of the collisions involve only two particles [6].

Table 2-1: Some of the possible reactions of plasma conditions in the plasma volume [6]

Reaction type	Name	
Electron impact	Ionisation	$e + A \rightarrow 2e + A^+$
	Dissociation	$e + AB \rightarrow e + A + B$
	Dissociative ionisation	$e + AB \rightarrow 2e + A + B^+$
	Dissociative attachment	$e + AB \rightarrow A^- + B$
	Electronic excitation	$e + A \rightarrow e + A^*$
	Ro-vibrational excitation	$e + AB \rightarrow e + AB\ (v, j \geq 1)$
	Momentum transfer	$e + A \rightarrow e + A$
Neutral	Dissociation	$AB + M \rightarrow A + B + M$
	Penning Ionisation	$Am + B \rightarrow A + B^+ + e$
	Atom transfer	$A + BC \rightarrow AB + C$
	Rearrangement	$AB + CD \rightarrow AC + BD$
	Recombination	$A + B + M \rightarrow AB + M$
	Energy transfer	$A^* + B \rightarrow A + B^*$
	Relaxation	$A^* + B \rightarrow A + B$
	Momentum transfer	$A + B \rightarrow A + B$
Ion	Neutralisation	$A^- + B^+ \rightarrow A + B$
		$A^- + BC^+ \rightarrow AB + C$
	Associative detachment	$A^- + B \rightarrow AB + e$
	Charge transfer	$A^+ + B \rightarrow A + B^+$
		$A^- + B \rightarrow A + B^-$
	Dissociative charge transfer	$A^+ + BC \rightarrow A + B + C^+$
	Momentum transfer	$A^+ + B \rightarrow A^+ + B$

Note that e represents electron, * an electronically excited state, v and j the vibrational and rotational quantum numbers, respectively, Am a long-lived metastable state and M indicates any molecule and indicates third-body stabilisation.

2-4-Plasma Application:

Plasmas underlie numerous important technological applications and devices as well as our understanding of much of the universe around us. Plasma processing technologies are of vital importance to several of the largest

manufacturing industries in the world. Foremost among these industries is the electronics industry, in which plasma-based processes are indispensable for the manufacture of very large-scale integrated microelectronic circuits. Plasma processing of materials is also a critical technology in, for example, the aerospace, automotive, steel, biomedical, and toxic waste management industries. Most recently, plasma processing technology has been utilized increasingly in the emerging technologies of diamond film and superconducting film growth [2].

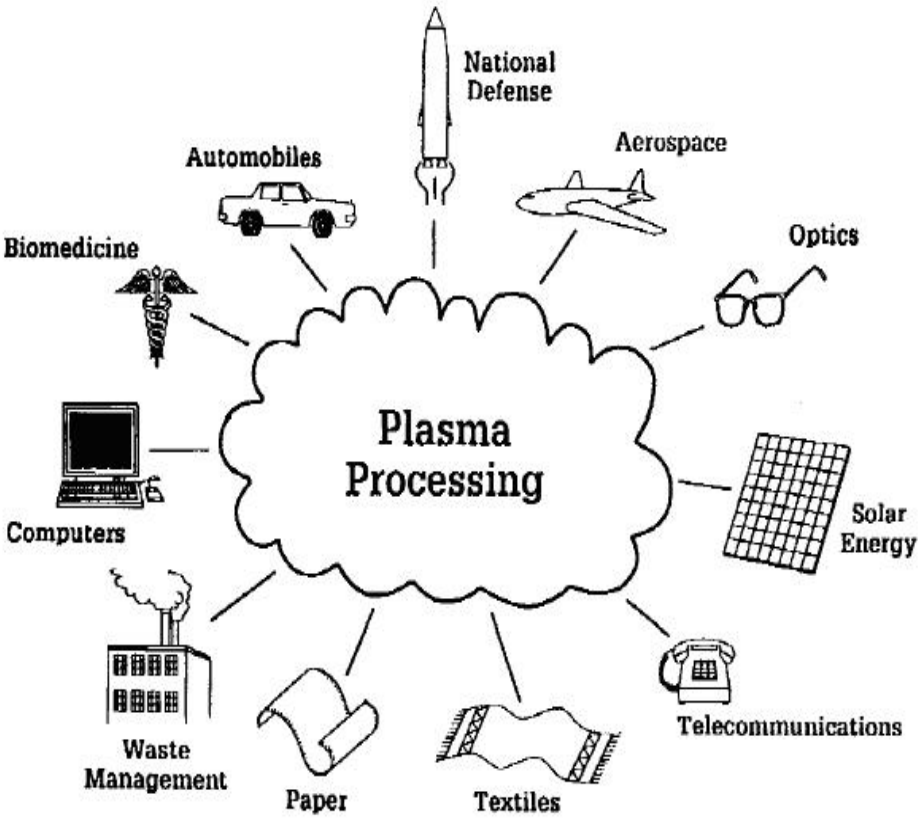

Figure 2-6: Plasma processing is a critical technology in many vital U.S. industries.

Researchers provide the foundation and underpinnings for present applications such as plasma processing of semiconductors, sterilization of some medical products, lamps, lasers, diamond coated films, high power microwave sources, and pulsed power switches. They also provide the foundation for important potential applications such as the generation of electrical energy from fusion and pollution control and removal of

hazardous chemicals. Plasma science encompasses a variety of science disciplines ranging from plasma physics to aspects of chemistry, atomic and molecular physics, and material science. Its broad, interdisciplinary nature also characterizes its plasma physics component, which includes ionized gases that range from weakly ionized to highly ionized, from collisional to collisionless, and from cold to hot. These terms characterize various plasmas ranging from relatively high-pressure gases with a small fraction of the atoms ionized and relatively low charged-particle temperatures -- for example, plasmas used in computer-chip processing and light sources -- to those in very low density gases with a large fraction of the gas atoms ionized and very high-temperature charged particles -- for example, fusion plasmas.

Different types of plasmas underlie different applications and different natural phenomena. However, many fundamental considerations span the broad parameter ranges that characterize the many natural and man-made plasmas that are important in our lives.

The diversity of what is included in "plasma science" makes the subject difficult to characterize. However, it is that same diversity that makes it such an important contributor to a wide range of applications and technological developments. Below is a list of just some of the many technological applications of plasmas:

Table 2-2. Industrial / Commercial Applications of Plasmas

Processing:
• Surface Processing
• Nonequilibrium (low pressure)
• Thermal (high pressure)

Volume Processing:
• Flue gas treatment
• Metal recovery
• Waste treatment

Chemical Synthesis:
• Plasma spraying
• Diamond film deposition
• Ceramic powders

Light Sources:
• High intensity discharge lamps
• Low pressure lamps
• Specialty sources

Surface Treatment:
• Ion implantation
• Hardening
• Welding
• Cutting
• Drilling

Propulsion

Flat-Panel Displays:
• Field-emitter arrays
• Plasma displays

Radiation Processing:
• Water purification
• Plant growth

Switches:
• Electric power
• Pulsed power

Energy Converters:
• MHD converters
• Thermionic energy converters

Medicine:
• Surface treatment
• Instrument sterilization

Isotope Separation

Beam Sources

Lasers

Material Analysis

2-5-Plasma Technologies for Textile Industry:

Using plasma technology to modify textile surfaces, etching, cleaning, polymerization and deposition reactions can be used to obtain nano-particle or nano-porous structures. The reactive plasma particles and radiation yield a nano-scaled interaction with material surfaces by chain scission and cross-linking reactions, radical formation, etching or deposition. The bulk properties of materials can thus be maintained. Moreover, it is a dry and eco-friendly technique, avoiding waste production as found in wet chemical processes. However, for the transfer into industry, both the feasibility of scale-up and

economic aspects have to be regarded [5, 6]. Some kinds of plasma technologies are discussed as bellow:

2-5-1-Plasma cleaning:

During textile manufacturing, sizing agent, mineral oils or acrylate-based spin finishes are applied, which form a film around the yarn or individual fibers to reduce friction and electrostatic charging. Due to the interaction of reactive plasma species, textile surfaces can be etched physically and chemically. Ion bombardment, radical density and VUV (vacuum ultraviolet) radiation determine the etching rate depending on reactor geometry, plasma excitation, gas type, flow, power, and pressure and textile material. Typical etch rates of organic materials are a few nanometers per second. Therefore, a plasma treatment can be used for cleaning of manufacturing residuals on textiles. Plasma cleaning is a prerequisite to obtaining good adhesion, e.g. to a subsequent plasma coating, and can be performed in a one-step process [6].

2-5-2-Plasma metallization:

For the metallization of textiles, sputter-based processes can be effectively used. Sputtering can be characterized as a non-equilibrium process at the energy range of interest for film deposition. Sputtering is initiated by the impact of energetic particles (in order of 100 eV) on a target material, e.g. a metal. The incident particle cause a multiatom kinetic collision process, whereby atoms near the surface may be dislodged with enough energy to overcome the surface binding energy and be emitted from the target. These atoms are known as sputtered atoms, and when they deposit on some other surface, the process is called sputter deposition. Inert gases such as argon are generally used for the sputtering of metals, whereas an addition of reactive gases (O_2, N_2, etc) yield reactive sputtering and e.g. metal oxide or nitrides. Moreover, alloys, ceramics, and polymers can be used as target materials. The energy of the incident particles can be enhanced by electrical and magnetic fields, while avoiding collisions. Therefore, we are mainly using magnetron sputtering with argon at a pressure of 1 Pa to obtain metallization on textiles and fibers [6].

2-5-3-Plasma modification:

Plasma modification of polymer surfaces may be categorized in to two major types of reaction:

Plasma treatment and plasma polymerization.

Advantageous of plasma processes include the following:

1. Modification can be confined to the surface layer without modifying the bulk properties of the polymer.
2. Exited species in gas plasma can modify the surfaces of all polymers, regardless of their structures and chemical reactivity.
3. By choice of the gas used, it is possible to choose the type of chemical modification for the polymer surface.
4. The use of gas plasma can avoid the problems in countered in wet chemical techniques such as residual solvent on the surface and swelling of the substrate.
5. Modification is fairly uniform over the whole surface.

The disadvantages of plasma processes are as follows:

1 Plasma treatments must be carried out in vacuum. This requirement increases the cost of operation.

2 The scale-up of an experimental setup to a large production reactor is not a simple process.

3 The plasma process is extremely complex.

In general, reactions of gas plasmas with polymers can be classified as follows:

1. Surface reactions: Reactions between gas-phase species and surface species and reactions between surface species produce functional groups and cross-links, respectively, at the surface. Examples of these reactions include plasma treatment by argon, ammonia, carbon monoxide, carbon dioxide, fluorine, hydrogen, nitrogen dioxide, oxygen, and water.
2. Plasma polymerization: The formation of a thin film on the surface of polymer via polymerization of an organic monomer such as CH_4, C_2H_6, C_2F_4, or C_3F_6 in plasma. It involves reactions between gas-species, reactions between gas-phase species and surface species, and reactions between surface species.

3. Etching: Materials are removed from a polymer surface by physical etching and chemical reactions at the surface to form volatile products. Oxygen plasma and oxygen and fluorine-containing plasmas are frequently used for the etching of polymers. [5]

2-5-4-Plasma Treatment:

A typical plasma system consists of a gas inlet, a reactor vessel, a vacuum pump, a matching network, and a power source. Various reactors have been used in plasma processing for dc and low-frequency glow discharges, internal electrodes are necessary.

A typical reactor is a bell jar with circular or square electrodes. As the frequency increases, electrodes maybe placed out side the reactor vessel. Plasma treatment has been used to improve print ability, wettablility, bond ability, biocompatibility, surface hardness, and surface heat resistance. It is also a means of cleaning polymer surfaces without a solvent and of introducing cross-linking at the surface.

The different types of gas or mixtures of gases that can be used for plasma treatment of polymers include argon, helium, hydrogen, nitrogen, ammonia, nitrous oxide, , oxygen, carbon dioxide, sulfur dioxide, water, and tetrafluoromethane. Oxygen and oxygen-containing plasmas are the most frequently and are very effective at increasing the surface energy of polymers. Nitrogen and nitrogen-containing plasmas are used to produce nitrogen functionality such as amino groups on polymer surfaces. Fluorine and fluorine-containing plasma are used to decrease the surface energy and to increase the hydrophobicity of polymer surfaces. Each gas produces a unique modified surface. In an oxygen plasma two process occur simultaneously: etching of polymer surface through the reaction of atomic oxygen with the surface carbon atoms, giving volatile reaction products; and the formation of oxygen functional groups at the polymer surface through the reactions between the active species from the plasma and the surface atoms. The balance of these two processes depends on the operation parameters of a given experiment.

Many parameters such as the nature of polymer substrate, the temperature of the substrate, electrode materials, pressure, power level, and gas flow rate play a significant role in affecting the outcome of a plasma treatment. [5]

2-5-5-Plasma Polymerization:

Thin polymer films with unique chemical and physical properties are produced by plasma polymerization. The films are prepared by vapor-phase deposition and can be formed on practically any substrate with good adhesion between the film and the substrate. Thin polymer films, which are usually highly cross-linked and pinhole free, have very good barrier properties. Such films find great potential in biomaterials applications and in the microelectronic industry.

Plasma deposited films have many special advantages:

1. A thin conformal film of thickness of few hundred angstroms to one micrometer can be easily prepared.
2. Films can be prepared with unique physical and chemical properties.
3. Films can be formed on practically any substrate, including polymer, metals, glass, ceramics, and semiconductors. In general, good adhesion between the film and substrate can be easily achieved.

The structure of plasma-deposited films is highly complex and depends on many factors, including reactor design, power level, substrate temperature frequency, monomer structure, monomer pressure, and monomer flow rate.

Polymer-polymerized films are generally amorphous cross-linked materials. The density of these films is higher than that of conventional polymer.

Low-energy surface polymer films can be prepared from monomers of fluorine-containing compounds, such as C_2F_4 and C_3F_6, and silicon-containing compounds, such as $(CH_3)Si$, $(CH_3\ O)_4Si$. High surface energy polymer films can be formed from oxygen-containing monomers, such as acrylic acid, acetone, methanol, formic acid, and allyl alcohol. The surface of a plasma-polymerized hydrocarbon film is more polar than its conventional counterpart because it contains a variety of oxygen functional groups that are due to the reactions of free radicals with atmospheric oxygen.

The adhesion of a plasma-polymerized film to a substrate is the most important factor in determining the success of plasma polymerization as a surface modification technique. In general, plasma-polymerized films adhere well to polymer substrates. The adhesion of a film to a substrate is determined by parameters such as deposition rate, thickness of

the film, and the nature of the substrate. Better adhesion is always achieved by a slower deposition rate, a small film thickness, and a polar polymer substrate. [5]

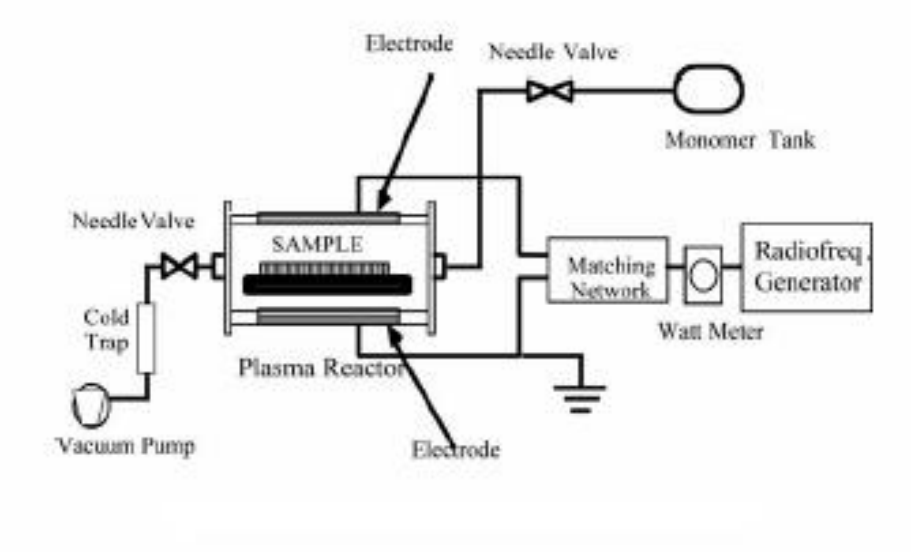

Figure 2-7: Schematic view of Plasma Polymerization setup. [8]

3-LITERATURE REVIEW

3-Applications of plasma in textile:

Several methods, based on physical and chemical modification of polymeric materials can be used, and one of the most important is the plasma treatment.

Previously, several wet treatments have been made, but the increase in industrial applications prompted the search for more profitable and environmentally clean process, such as low pressure plasma techniques. [9, 10]

3-1-Rakowski's opinion about advantages of plasma treatment of textiles.

The practical advantages of textile plasma exposure have been documented by Rakowski [10]. Who compared two processes used to achieve printable wool tow. One was a conventional chlorination process and the other a new process based on the exposure of wool tow to low pressure plasma.

Wool tow is fed continuously into a vacuum chamber operating at pressure of 2 to 6 torr, where glow discharge plasma provides active species to which the wool exposed. A novel feature of this approach is the continues feeding of the wool tow into and out of the vacuum system through several differential stages of vacuum pumping.

Rakowski compared this low pressure plasma surface treatment with conventional chlorination and found that plasma modification of wool saves large quantities of water, chemical, and electrical energy. Compared with conventional chlorination, the low pressure (2-6 torr) plasma modification of 120 ton/year of wool saves 27000 m^3 of water, 44 ton of sodium hypochlorite, 16 ton of sodium bisulphate, 11 ton of sulphuric acid, and 685 MWh of electrical energy. These large savings are possible since the plasma process dose not produce large volumes of waste or toxic byproducts.

Whether at one atmosphere or at low pressure plasmas achieve their surface treatment effects as a result of the interaction of one or more active species

from the plasma with the surface of interest. These active species are more chemically reactive and more energetic than the species associated with conventional chemical processing. Such species may include ultra chemical processing. Such species may include ultra violet photons, which are capable of breaking chemical bonds, and photons in the visible part of the spectrum, which can produce a positive surface charge by the

photoelectric effect. Charge particles are a second major class of active species from plasma; they include electrons that either recombine on the surface or build up a surface charge; ions that may be produced by ionization events, attachment, or charge exchange in the plasma; and free radicals or other charged molecular fragments such as OH resulting from plasma chemical reactions. A third major class of active species is neutral particles, which can include very reactive atoms such as monatomic fluorine, oxygen, or other atomic fragments; atoms or molecules in excited atomic stages; and highly reactive molecular fragments, including monomers produced in the plasma. [10]

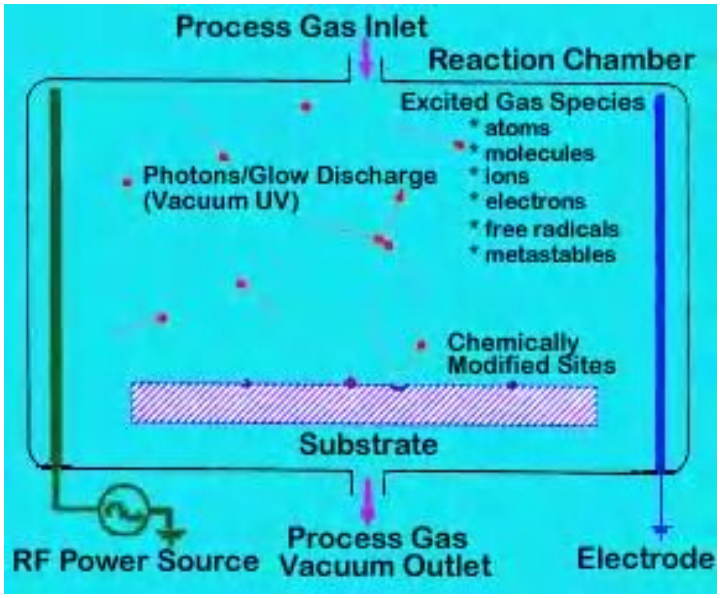

Figure 3-1 : Schematic diagram of plasma setup.

3-2-Self-cleaning of wool-polyamide and polyester textiles by TiO2-rutile modification under daylight irradiation at ambient temperature

This study presents radio frequency plasma (RF-plasma), microwave plasma (MW plasma), and vacuum-UV light irradiation as pretreatment of synthetic textile surfaces allowing the loading of TiO_2 by wet chemical techniques in the form of transparent coatings constituted of nanoparticles of diverse sizes. These loaded textiles show a significant photo-oxidative activity under visible light in air under mild conditions, which discolors and mineralizes persistent pigment stains contained in wine and coffee. The mineralization of stains on the textile loaded with TiO_2 was monitored

quantitatively to assess the appropriate surface pretreatment in conjunction with the most suitable deposition method of TiO_2 colloids, powders, or combination of both. Their photocatalytic activity allowed, in kinetically acceptable times, the almost complete discoloration of coffee and wine stains. The observed discoloration of colored stains seems to involve visible light sensitization of the stain pigment on the TiO_2-loaded textile. The size of the particles obtained from colloidal precursors of TiO_2 varied between 5 and 25 nm.

This study aims to increase the bondability of TiO_2 on wool-polyamide and polyester textiles by surface textile modifications induced by RF-plasma, MW-plasma and UV irradiation.

Different pretreatment methods of synthetic textiles have been studied that allow the TiO_2 coatings to discolor wine and coffee stains under visible light in reasonable time. Posttreatment at temperatures of 100 ˚C or less were shown to be sufficient to attach TiO_2 to the synthetic textiles. The nano-particles of TiO_2 remain fairly stable on the textile surface after the photochemical discoloring of stains. A combination of TiO_2 powder and TTIP Titanium tetra-isopropoxide (20 ml) colloids deposited on wool-polyamide or polyester textiles showed to be kinetically suitable for the self-cleaning of wine and coffee stains using a solar simulated light with 50% AM1. Under neon light the self-cleaning effect needed much longer times [11].

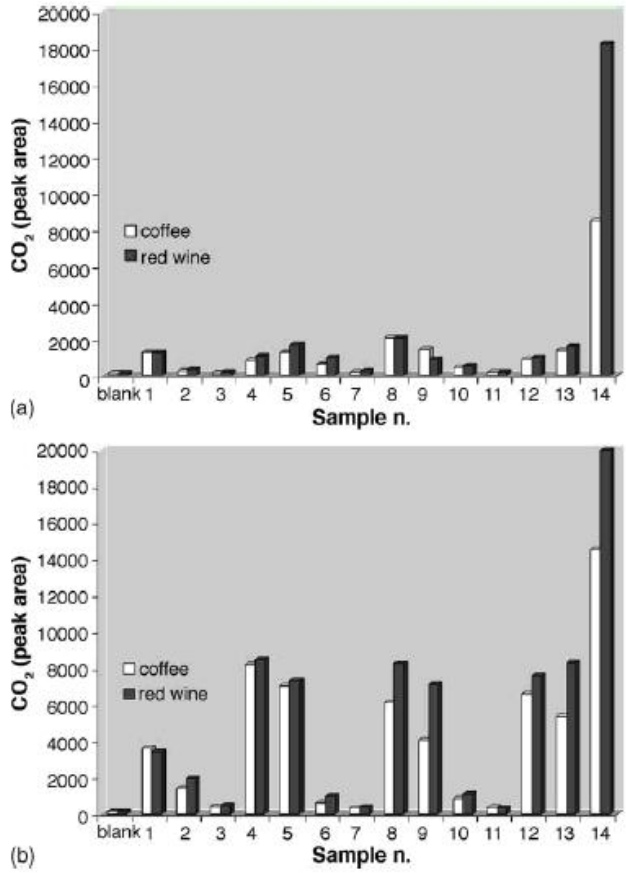

Figure 3-2. CO_2 produced during the photocatalytic degradation of stains of coffee and red wine on TiO_2-loaded polyester (a) and wool-polyamide (b). Irradiation time: 24 h. During this study a Sun test sunlight simulator was used with a power of 50 mW/cm^2 [11].

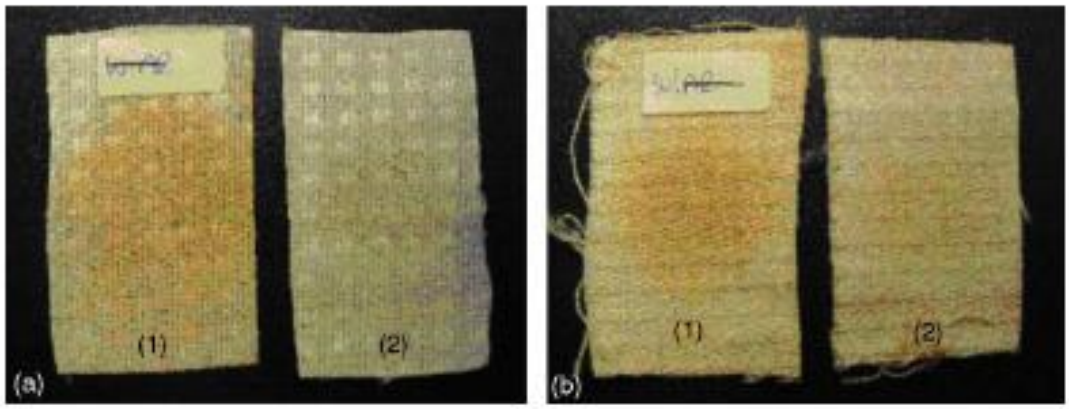

Figure 3-3. Discoloration of red wine stains on the sample 14. (a) Polyester fabrics, (b) wool-polyamide. Textile before irradiation (1) and after 24 h Sun test irradiation (2) [11].

3-3-Plasma enhanced CVD deposition of titanium oxide for biomedical applications:

Radio frequency plasma enhanced chemical vapor deposition (RF PECVD) of titanium oxide films for bactericidal applications was reported. Titanium tetrachloride was used as a precursor compound, and the depositions were performed in the presence of oxygen. The films were deposited on glass and cotton textile substrates. Optical properties, and namely refractive index and extinction coefficient, of the films deposited on glass were used as criteria for their quality. Bactericidal properties were studied using cultures of K12 strain of Esterichia coli and the ultraviolet light C (UV-C) irradiation. An effect of an RF power of the discharge on both the optical and the bactericidal properties of the films was investigated. The results showed a substantial enhancement of the bactericidal activity of UV irradiation for the surfaces modified with the presented process. A strong correlation between the bactericidal efficiency of the films and their refractive index has been observed. The morphology and elemental composition of the coatings applied to cotton textile was also investigated using electron scanning microscopy (SEM) and energy dispersive X-ray (EDX) technique.

As a source of titanium, liquid $TiCl_4$ has used. Its vapor has supplied from a bubbler using a stream of argon. The flow rate of $TiCl_4$ vapor was a function of the temperature of liquid precursor and of the flow rate of argon. $TiCl_4$ vapor, carried by argon, was introduced to the reactor's main supply manifold where it was mixed with oxygen. RF power input was used as an operational parameter of deposition and it was varied in the range between 20 and 300 W. Microscope cover glass slides of 20×20 mm^2 dimensions and 50×50 mm^2 pieces of cotton knitwear were used as substrates. They were placed on the lower, RF powered, electrode.

The results presented show that a PECVD produced titanium oxide coatings exhibited to a large degree photo catalytic properties, revealed by a substantially enhanced bactericidal activity. [12]

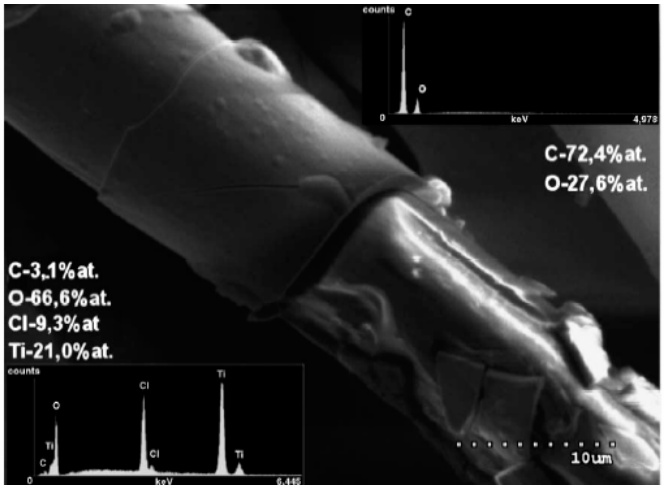

Figure 3-4: A scanning electron micrograph of a single cotton fiber, deposited with titanium oxide film (at the RF power of 100 w) and partially of the coating. The windows present EDX results for coated and uncoated fragment, respectively. [12]

3-4-Plasma-induced graft-polymerization of flame retardant monomers onto PAN fabrics

Tsafaka et all, presents the use of the low-pressure plasma technique to confer a fire-resistant character to polyacrylonitrile (PAN) textiles. He investigated the argon plasma-induced graft-polymerization of four acrylate monomers containing phosphorus, diethyl(acryloyloxyethyl)- phosphate (DEAEP), diethyl-2(methacryloyloxyethyl)phosphate (DEMEP), diethyl (acryloyloxymethyl) phosphonate (DEAMP) and dimethyl (acryloyloxymethyl) phosphonate (DMAMP), which are known to be effective monomers for the fireproofing of polymeric substrates in classical polymerizations. The grafting and the polymerization processes taking place on the surface of the PAN textile were followed by weighing measurements, IR(ATR) spectroscopy and SEM. The fire retardant character of the treated fabrics was investigated by thermo gravimetric analyses and LOI measurements. Furthermore, they were able to prove the persistence of the coatings towards several washing cycles by using the accelerated method of laundering proposed by McSherry and al. [13]

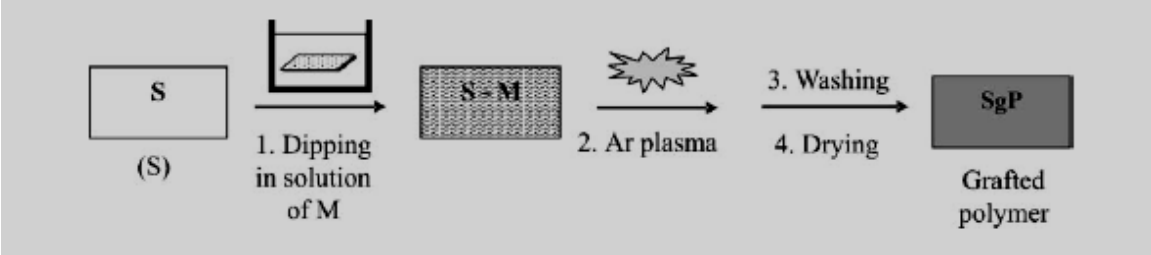

Figure 3-5: Experimental procedure for the Ar-plasma-induced graft polymerization of monomers. [13]

All the experiments were performed using the protocol as follows: In a first step, pieces of PAN fabrics were cut (52 × 140 mm) and then immersed at room temperature for 1 min in an ethanolic solution containing 200 g/L or 300 g/L of the monomers (DEMEP, DEAEP, DEAMP or DMAMP) in the presence of 5% (w/w) of the photoinitiator (BAPO) and various amounts (0, 10 and 20% (w/w)) of the crosslinking agent (EGDA).These impregnated fabrics are then pressed to evacuate the excess of the solution, placed onto glass plates and submitted to a MW argon plasma (FAr=125 sccm, base pressure=40 Pa, P=100 W, t=15 min). After this treatment, the fabrics were washed with ethanol, then with water in a soxhlet apparatus, and air-dried at room temperature for 2 days.

It was seen in Table 1, that, by grafting acrylate containing phosphorus, very good flame retardant properties were achieved on the surface of PAN fabrics [13].

Table 3-1: LOI values of treated PAN fabrics as a function of monomer type, monomer concentration and the percentage of grafting after washing and air-drying

	[Monomer], g/L	EGDA, % (w/w)	Grafting, %	Measured P content on PAN, %	LOI
DEMEP	200	20	20.56	1.6	22
DEAEP	200	20	24.14	2	22
DEAMP	200	20	22.5	–	22.5
	300	20	28.5	2.7	23.5
DMAMP	200	10	20.11	2.3	23.5
	200	20	28.5	3	24.5
	300	20	39.8	3.9	26.5

3-5-Low temperature plasma-treated nylon fabrics

Nylon 6 fabrics were treated with low temperature plasma (LTP) with three non-polymerizing gases: (i) oxygen, (ii) argon and (iii) tetralluoromethane. After plasma treatment, the properties of the fabric, including surface morphology, low-stress mechanical properties, air permeability and thermal properties, have investigated. The nylon fabrics treated with different plasma gases exhibited different morphological changes. Low-stress mechanical properties obtained by means of the Kawabata evaluation system fabric (KES-F) revealed that the surface friction, tensile, shearing, bending and compression properties altered after the treatments. The changes in these properties are believed to be related closely to the inter-fiber/inter-yarn frictional force induced by the LTP treatment. The air permeability of the samples was studied using a KES-F8-AP1 air permeability tester. The air resistance *(R)* was recorded in term of kPa s/m: a larger value of air resistance *(R)* indicates poorer air permeability of the fabric and vice versa. A slightly decrease in the air permeability of the treated fabrics was found which is probably due to plasma action effecting on increase in the fabric thickness and a change in the fabric surface morphology. The change in the thermal properties of the treated fabrics was in good agreement with the above findings and can be attributed to the amount of air trapped between the yarns. This experimental work suggests that the changed properties induced by LTP can effect an improvement in certain textile products [14].

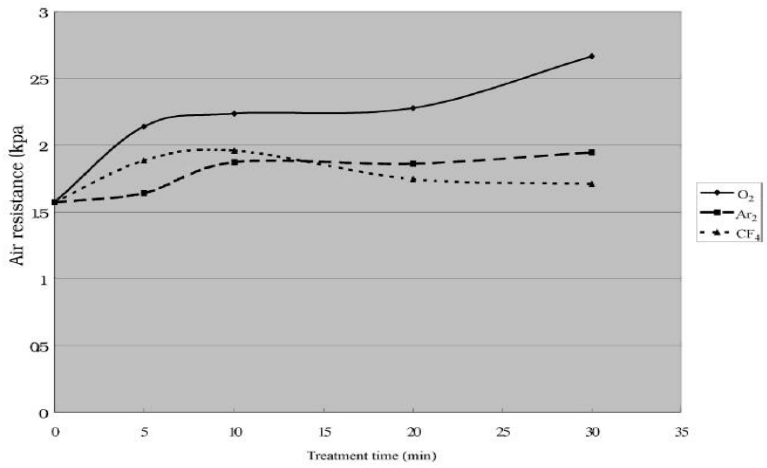

Figure 3-6: The air resistance of nylon fabric vs LTP treatment

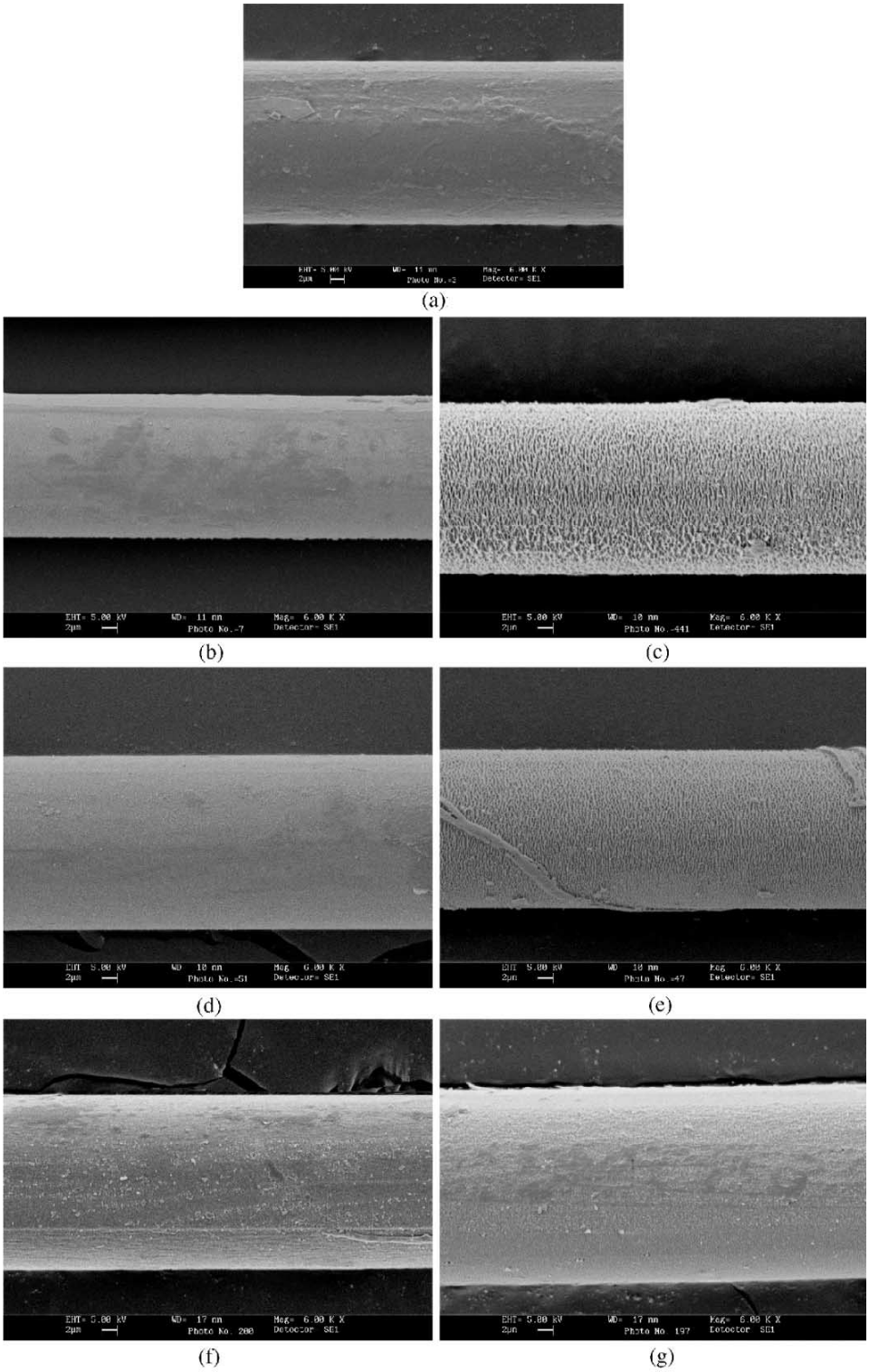

Figure 3-7 : SEM photographs of LTP treated Nylon 6 (a) untreated, (b) 5 min O_2 (c) 30 min O_2 (d) 5 min Ar, (e) 30 min Ar, (f) 5 min CF_4, (g) 30 min CF_4 [14].